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DETERMINATION OF THE ULTRASONIC PROPERTIES OF  
COMPOSITES

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## INTRODUCTION

This end-of-year report summarizes the progress made on NASA Grant NAG-1-431 (*The Determination of the Ultrasonic Properties of Composites*) from December, 1983, to November, 1984. The research effort described here encompassed two facets of the propagation of ultrasonic waves in composite structures. This report will include a section on the imaging of delaminations and a section on the temperature dependence of the acoustical properties of composites.

### I. Imaging Delaminations in FWC Solid Rocket Motors.

To provide quantitative nondestructive evaluation (NDE) of composite structures, two facets must be examined. First, the interaction between the ultrasonic wave and the material must be clearly understood and second, any measured quantity presented as a means of evaluating damage must appear in a manner that allows easy interpretation of the damage. We have been engaged in an effort to examine these two research areas and the next section will review our progress.

At the start of the grant period, a research opportunity arose for the study of thick-walled filament wound casing (FWC) structures. Hercules, Inc., of Magna, Utah, is constructing the FWC solid rocket motors for the space shuttle. A solid rocket motor of this size has never been constructed before and the quantitative NDE of such a structure is just being developed by both industry and government laboratories. One of the mechanisms of immediate importance to the NASA effort is the quantitative technology for imaging delaminations.

It is necessary to image delaminations in FWC structures and develop a reject/accept criteria based on the anticipated effect of the delamination on performance.

The FWC structure is formed by winding a filament onto a mandrel. The filament is under tension and the orientation to the cylinder axis is varied to give radial and hoop ply directions. In the final configuration the cylinder is twelve feet in diameter and has a skin thickness of about 1-3/4 inches. The cylinder is undergoing partial cure at room temperature while the winding proceeds over the course of tens of hours. Upon completion of winding, the cylinder is placed in an oven to complete the cure process.

A delamination may be caused by internal residual stresses, contamination, or improper cure. It is possible for a structure of this size that compressive loads produced by the manufacturing process can be of sufficient magnitude to close the delamination with respect to a propagating acoustic wave. To investigate this possibility, we performed a series of experiments which modeled a typical technique for imaging delaminations. A technique which is prevalent is to compare the C-scan signal level of a reference

block to that of the specimen. The receiving electronics are adjusted for complete saturation with the reference block which is designated to be free of anomalies. When scanning the specimen, a trigger is set for a signal level -40dB down from that of the reference block. Any signal below this level will be designated a delamination. Thus, the objective of these experiments was to examine the effect of pressure on the acoustical signal and note its relation to a reference standard.

The samples used were prepared from FWC material which was 1-3/4 inches thick and in blocks 1-1/2 inches on a side. One sample was kept as a reference block and four others were cut through the thickness by a diamond saw. This provided the worse case pseudo-delamination.

The experimental arrangement is shown in figure 1. The sawn samples are placed between transducer holders which provide constant spring tension on the transducers while force is applied to the sample. The broad-band 1MHz transducers are bonded to the samples with propylene glycol and driven with a pulsed oscillator. Tone bursts of 70 cycles at 720KHz were amplified by a 40-watt power amplifier to drive the sending transducer. The received signal, amplified by a wideband 20dB gain preamp, is input to a spectrum analyzer to determine the signal strength at resonance.

Figure 2 shows some representative data. The reference standard gave an indication of -28dB and the dotted line in the figure represents the signal level 40dB down from this reference. Thus, using the proposed criteria for delamination, any signal less than the 40dB line is indicative of a delamination. The results of compressive loads shows that, at a pressure of 47 pounds per square inch, this sample appears (by the -40dB criteria) to be free of delamination.

Thus, for a worse case situation, it is possible to mask the acoustical indication of a delamination. This is a distinct possibility for a structure that is wound under tension and is of substantial thickness.

This introduces questions about the reliability of any NDE technique based only on loss of signal (-40dB). A possible remedy could include multiple C-scan passes with varying dynamic range settings for the receiving electronics or using wider dynamic range technologies.

## II. Temperature Dependence of the Acoustical Properties of Composites

The results of this effort were presented at the 1984 Ultrasonics Symposium, and the submitted article for the proceedings of that meeting is included here.

ORIGINAL PAGE  
OF POOR QUALITY

TEMPERATURE DEPENDENCE OF THE ACOUSTICAL PROPERTIES OF COMPOSITES

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ABSTRACT

Ultrasonic attenuation in graphite fiber-reinforced polymer composites is the result of two mechanisms: absorption by the matrix and scattering by the fibers and defects. A technique is described for experimentally separating the absorption contribution from the scattering contribution by measuring the temperature dependence of the acoustic properties of the composite. As the temperature of the composite decreases, the matrix attenuation changes appreciably, while energy loss by scattering remains relatively constant. Ultrasonic data is presented on a fiber-reinforced polysulfone composite and contrasted to data for a polysulfone plate.

I. Introduction

The integration of composites into critical components of aerospace structures depends on being able to nondestructively evaluate their integrity. Conventional ultrasonic techniques have been unable to detect critical flaws such as broken fibers and poor matrix-fiber bonding. Detection of these flaws will require new ultrasonic techniques which take advantage of the unique properties of composites. Development of these techniques requires a better understanding of the acoustic properties of composites.

A composite of great interest for aerospace applications is a lamination of graphite fiber arranged in different directions and reinforced with a polymer matrix. The acoustic properties of the composite depend on the acoustic properties of the matrix, fiber and matrix-fiber interface and the direction of the propagation in the composite. The matrix is a viscoelastic solid with a low acoustic velocity and high acoustic attenuation. The fiber is a polycrystalline solid with a very high acoustic velocity and low attenuation. The acoustic properties of the matrix-fiber interface are unknown.

For waves propagating perpendicular to the fiber direction, one expects the velocity to be increased over the velocity of the matrix as a result of the reinforcing of the fiber. The attenuation in a defect-free composite is expected

to result from absorption by the matrix, scattering from the fibers and an absorption by the matrix-fiber interface material. The matrix properties may be separated from the fiber properties in one of two ways. The first technique involves measuring the acoustic properties as a function of frequency as done by Williams, Lee, and Nabye-Hashemi [1]. The second technique, which we present here, takes advantage of the viscoelastic character of the matrix, and matrix-fiber interface. For a viscoelastic solid, as its temperature changes, dramatic changes occur in its attenuation caused by absorption. In contrast, the scattering component should remain approximately constant as the temperature changes. By measuring the temperature dependence of the attenuation of the resin and composite, a data base is generated for comparison of different models for acoustic propagation in composites.

The experimental technique for measuring the acoustic response of the matrix and composite as a function of temperature is summarized in section II. Section III presents a technique for reducing the acoustic response to the acoustic properties of the sample. In section IV, we report on measurements made at a series of different temperatures. A discussion of the results is presented in section V.

II. Experimental Technique

The experimental arrangement is shown in figure 1. A glass buffer rod acoustically couples the sample to the transducer, with Nonaq grease acting as the coupling agent between sample and buffer rod, and transducer and buffer rod. The samples are flat plates, with a thickness chosen to keep the acoustic transient time less than half the transient time in the buffer rod. A single cycle of a 5-MHz sine wave is used to excite the 1/2-inch diameter 5 MHz transducer. Measurements of the waveform are digitized at a sample rate of 100 MHz signal averaged 200 times and stored on computer. A typical response is shown in figure 2.

The transducer-buffer rod-sample configuration is placed inside the interior chamber of a two-chamber insulated box. Liquid nitrogen is placed in the exterior chamber and allowed to slowly boil off. A chromel-alumel thermocouple monitors the temperature of the sample. At 15-minute intervals

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the acoustic response and temperature of the sample are measured.

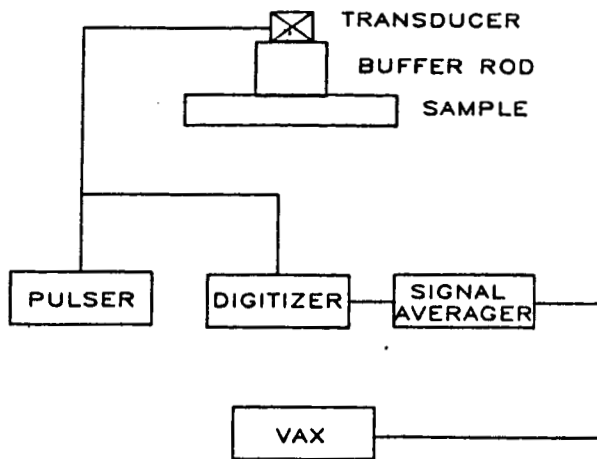


Fig. 1 Experimental arrangement.

### III. Data Reduction

The acoustic response of the sample needs to be reduced to determine the acoustic properties of the sample. The waveform is analyzed using a variation of a technique of Papadakis, Fowler, and Lynnworth [2]. The acoustic response as seen in figure 2 is a result of the reflection from the

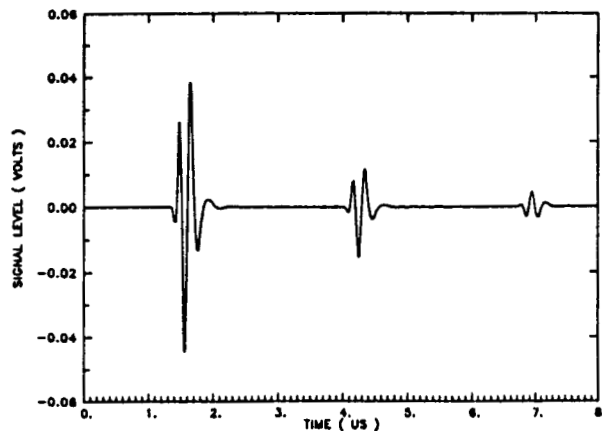


Fig. 2 Acoustic response of buffer rod/polysulfone sample at  $-20^{\circ}\text{C}$ .

sample-buffer rod interface and the first and second echoes from the back surface of the sample. The reflection from the interface between the buffer rod and the sample is used to fit the echoes from the back surface of the sample using the expression

$$\frac{(1 - R^2)e^{-2d(K_1 + K_2\omega) - 2i\omega d/c}}{R} - \frac{R^2(1 - R^2)e^{-4d(K_1 + K_2\omega) - 4i\omega d/c}}{R} \quad (1)$$

where  $R$  is the reflection coefficient,  $d$  is the thickness,  $c$  is the longitudinal velocity,  $K_1$  and  $K_2$  give the frequency dependence of the attenuation, which is assumed to be a constant plus a linear frequency dependent term. This expression is convolved with the echo from the buffer rod-sample interface and used as the fitting function in a least squares routine using  $R$ ,  $c$ ,  $K_1$  and  $K_2$  as independent variables. It should be noted here that this technique assumes the bond thickness is thin enough to cause a negligible error. If this is not the case it should show up in an inability to fit the data with equation (1).

### IV. Results

The acoustic response as a function of temperature was measured for a composite plate consisting of graphite fiber and a polysulfone matrix. The composite is a 50-ply laminated plate with 0- and 90-degree alternating fiber orientations. The thickness of the composite plate was 6.2 mm. The fit of the response using the technique detailed in the previous section is shown in figure 3. As

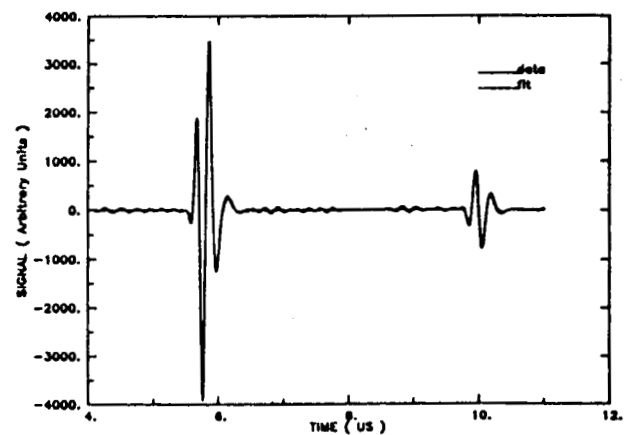


Fig. 3 Experimental data (—) and fit (---) using Eq. (1) for the composite sample at  $-20^{\circ}\text{C}$ .

can be seen from the figure, the fit is quite good so that the acoustic properties determined from the fit should be accurate.

For comparison, the acoustic response was measured as a function of temperature for a polysulfone plate. The polysulfone was obtained from 3-mm thick sheet stock. Its measured response was also

analyzed to determine its acoustic velocity and attenuation using the technique described previously.

A comparison of the change in velocity as a function of temperature for the polysulfone matrix and the composite are shown in figure 4. As

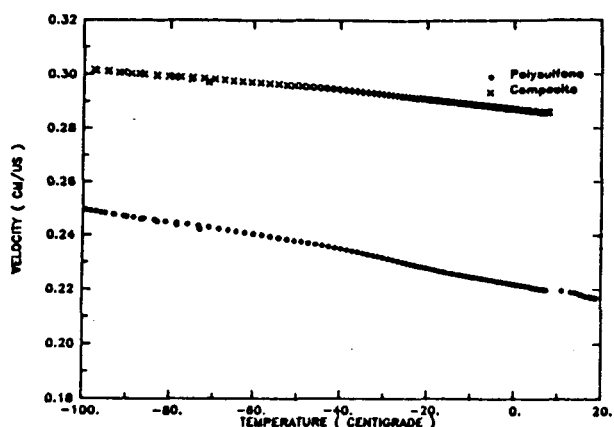


Fig. 4 Temperature dependence of velocity for polysulfone (O) and the composite (X).

expected the composite has a higher velocity than the matrix resulting from the reinforcement of the fibers. Both samples show an increase in the velocity as the temperature decreases, which is characteristic of a viscoelastic solid. A thickness correction for thermal contraction is not included, since the coefficients of thermal expansion are not known for the composite and the matrix.

Figure 5 shows a comparison of the attenuations as a function of temperature for the matrix and the

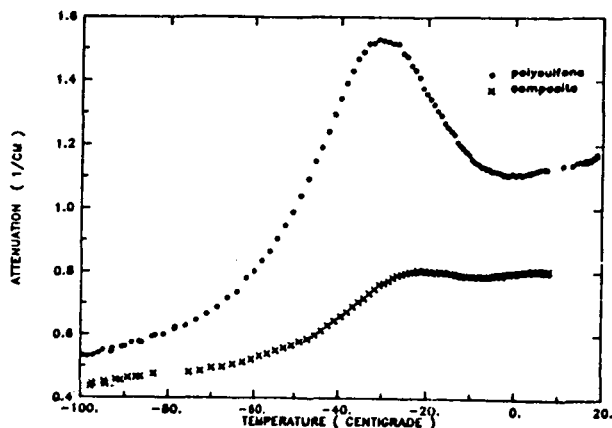


Fig. 5 Temperature dependence of attenuation for polysulfone (O) and the composite (X).

composite. The matrix has a relaxation maximum at approximately  $-35^{\circ}\text{C}$ . The composite has a similar peak at  $-25^{\circ}\text{C}$ . This offset may be caused by three sources. First, it may be a result of the experimental error in the measurement. Second, the matrix material is dissolved in solvent before applying it to the fiber; the shift in the relaxation peak may be the result of residual solvent which remains after the fabrication of the composite. Third, the shift may result from a viscoelastic matrix-fiber interface material, which may contribute to the relaxation mechanism.

A simple two-component model can be used to relate the attenuation of the matrix to the attenuation of the composite. The model assumes that only the matrix dominates the absorption component of the attenuation while the scattering component is temperature insensitive. The total attenuation in the composite is factored into two components

$$\sigma_c = \sigma_{\text{matrix}} * V_f + \sigma_s,$$

where  $V_f$  is equivalent to a volume fraction of the matrix and  $\sigma_s$  is the scattering contribution to the attenuation.  $V_f$  and  $\sigma_s$  are varied to give the least squares fit of the experimentally-measured temperature dependence of the attenuation of the composite shown in figure 6. The value

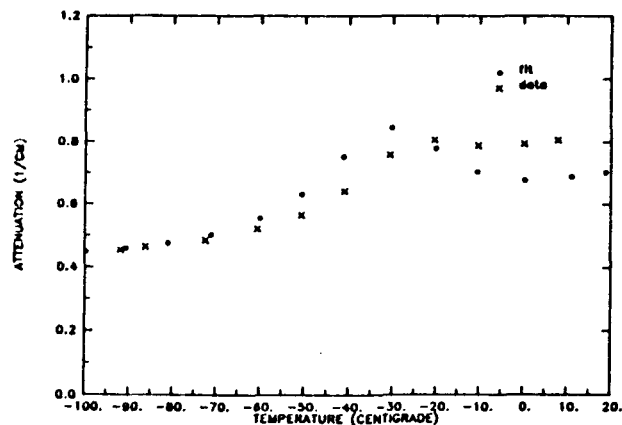


Fig. 6 Temperature dependence of the attenuation of the composite (X) and a fit (O) using Eq. (2).

found from this data for the volume fraction of the matrix is 40 percent which is in the range of 30-50 percent for composites. This also suggests the scattering contribution to the attenuation is 0.23 (1/cm) which is about a third of the room temperature attenuation. The model does not give a perfect fit of the data, but illustrates how the temperature data can be used to check different models for the acoustic properties of composites.

#### V. Conclusion

In this study we have demonstrated a technique for experimentally separating the contribution of scattering to the attenuation from the attenuation resulting from the viscoelastic absorption. The technique measures the attenuation of the matrix material and the composite as a function of temperature. The change in the attenuation from viscoelastic absorption has a dramatic temperature dependence, while the scattering contribution has little temperature dependence. The resulting data can be used to evaluate different models for acoustic propagation in composites.

#### References

- [1] J. H. Williams, Jr., S. S. Lee, and H. Nayeb-Hashemi. Jour. of Nondestructive Evaluation, 1, 191-199 (1980).
- [2] E. P. Papadakis, K. A. Fowler, and L. C. Lynnworth, Jour. Acous. Soc. Am., 53, 1336-1343 (1973).

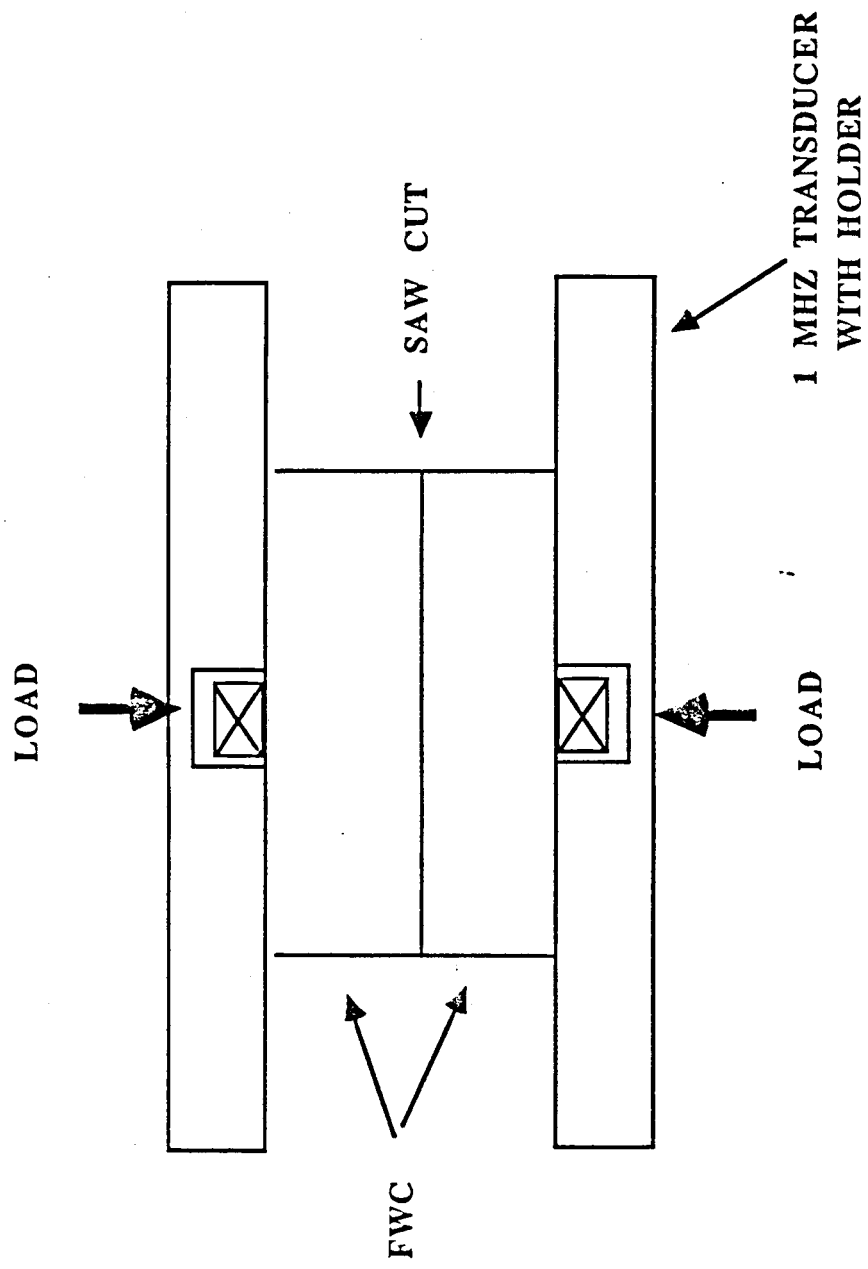


FIGURE 1.



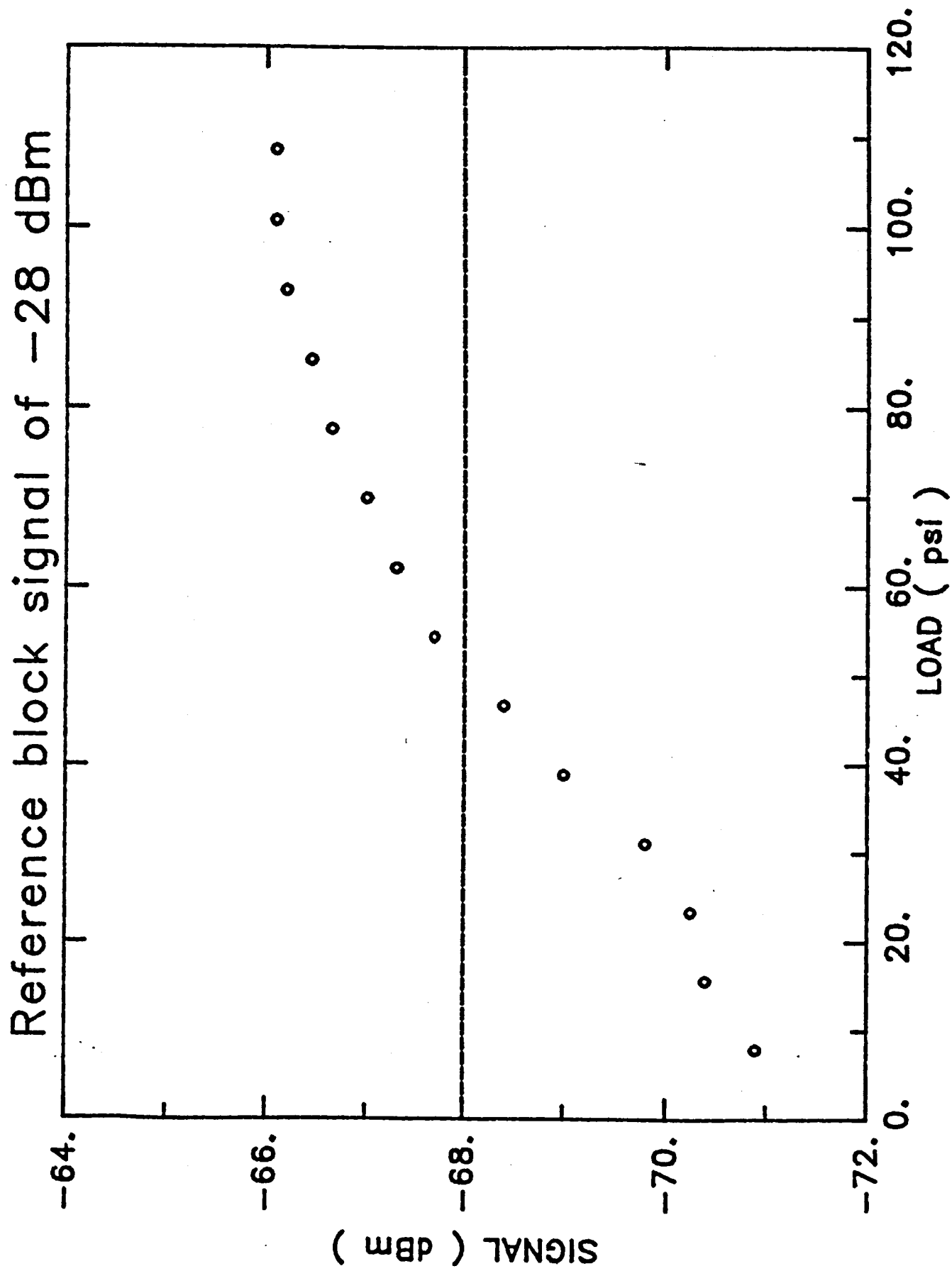


Figure 2.